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Optimizing the torrefaction of mixed softwood by response surface methodology for biomass upgrading to high energy density

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ABSTRACT

The optimal conditions for the torrefaction of mixed softwood were investigated by response surface methodology. This showed that the chemical composition of torrefied biomass was influenced by the severity factor of torrefaction. The lignin content in the torrefied biomass increased with the SF, while holocellulose content decreased. Similarly, the carbon content energy value of torrefied biomass ranged from 19.31 to 22.12 MJ/kg increased from 50.79 to 57.36%, while the hydrogen and oxygen contents decreased. The energy value of torrefied biomass ranged from 19.31 to 22.12 MJ/kg. This implied that the energy contained in the torrefied biomass increased by 4–19%, when compared with the untreated biomass. The energy value and weight loss in biomass slowly increased as the SF increased up until 6.12; and then dramatically increased as the SF increased further from 6.12 to 7.0. However, the energy yield started decreasing at SF value higher than 6.12; and the highest energy yield was obtained at low SF.

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1. Introduction

The development of sustainable technologies for the production of environmentally friendly fuels is warranted due to the acceleration of global warming and depletion of the petroleum in the world. Among biomass, lignocellulosic biomass plays a major role in the production of sustainable energy, because it is renewable, abundant, relatively inexpensive, and often locally available. In recent years, growing attention has been focused worldwide on the use of lignocellulosic biomass as a feedstock to produce biofuel pellet as an alternative to fossil fuel (Nilsson et al., 2011; Stahl and Berghel, 2011). The main advantage of wood pellet, in comparison to unprocessed wood, is the higher energy density and therefore a lower transport and storage costs. In addition, wood pellet has low ash content, as compared to agricultural biomass such as rice straw and wheat straw, implying a lower risk of corrosion and fouling (Jenkins et al., 1998; Obernberger and Thek, 2004). However, wood pellet has relatively high moisture content, low energy density, hydrophilic behavior, and storage issues when compared with coal (Chen and Kuo, 2011). Also, wood pellet does not have a constant energy value and ash content because pellet can be produced from various resources. Torrefaction of lignocellulosic biomass was, thus, introduced as a method to dispel these disadvantages (Chen and Kuo, 2011; Repellin et al., 2010; Turner et al., 2010).

Torrefaction is a thermal pretreatment process where raw material is heated in an inert or nitrogen atmosphere at a temperature of 200–300 °C (Prins et al., 2006). This provides a hydrophobic condition for the biomass due to the removal of the hydroxyl group during thermal treatment (Bourgeois et al., 1989). Therefore, torrefied biomass provides suitable chemical and physical characteristics for long-distance transportation and long-term storage. Additionally, the advantages of torrefaction include a higher energy value or energy density, lower O/C ratio and moisture content, and better grinding (Phanphanich and Mani, 2010). During the torrefaction of biomass, most of the volatile compounds are removed from the biomass as vapors, and consequently results in higher energy density. Many researchers have reported that torrefaction temperature for lignocellulosic biomass range from 250 to 300 °C (Chen and Kuo, 2011; Phanphanich and Mani, 2010; Prins et al., 2006; Stahl and Berghel, 2011). In addition, Chen and Kuo suggested that biomass subjected to light torrefaction (less than 1 h) is appropriate for producing solid fuels with a higher energy density, as compared to unprocessed biomass (2010).

However, torrefaction of biomass results in biomass weight loss depending on the torrefaction condition. A high energy value of torrefied biomass is indicative of such weight loss during torrefaction. Therefore, torrefied biomass should be evaluated in terms of energy yield. In this study, the optimal condition for torrefaction to obtain a high energy yield of torrefied biomass was investigated by response surface methodology.

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2. Methods

2.1. Materials

Mixed softwood chips were purchased from Poong Lim Inc. (Daejeon, Korea). Wood chips were screened to the size of 10–30 mm, using sieves (9.5 and 31.5 mesh) and dried to below 10% moisture content for safe outside storage.

2.2. Torrefaction process

The torrefaction conditions were based on a 2^2 factorial design with star design (four axial points) and three replicates in the central point (Table 1). The wood chips were dried at 105 °C for 24 h before torrefaction in order to remove the water remaining in the biomass. The dried wood chips (500 g) were placed in a batch reactor (designed by Drying Engineering Inc., Korea) which has a temperature controller, and which was sealed to increase the temperature. The wood chips were torrefied under nine different conditions with stirring. Torrefaction was performed under anoxic condition to avoid oxidation and ignition. A nitrogen flow of 2 L/min was used as the inert carrier gas, and an exhaust gas line was passed through a series of condensers to collect condensable compounds generated from the wood chips during torrefaction. A schematic diagram of the torrefaction reactor is shown in Fig. 1.

Table 1
The 2^2 factorial design with four axial points and three replicates in the central point matrix employed for two independent variables.

Sample No.	Variables		Coded levels		Severity factor (SF)
	Time (min) X_1	Temperature (°C) X_2	Time x_1	Temperature x_2	
1	50	250	0	0	6.12
2	50	250	0	0	6.12
3	50	250	0	0	6.12
4	70	270	1	1	6.85
5	70	230	1	−1	5.67
6	30	270	−1	1	6.48
7	30	230	−1	−1	5.30
8	80	250	1.4	0	6.32
9	50	280	0	1.4	7.00
10	20	250	−1.4	0	5.72
11	50	220	0	−1.4	5.23

The condensed liquid was analyzed by gas chromatography and mass spectrometer (GC/MS, Agilent 6890 and 5973, USA). After torrefaction, the heater was turned off, and the reactor was left to cool down to room temperature.

For the torrefaction of biomass, a severity factor (SF) was used to integrate the effects of reaction times and temperature into a single variable (Lloyd and Wyman, 2005). The SF used in this study is defined as

$$\mathrm{SF} = \log\{t \cdot \exp[(T_{\mathrm{H}} - T_{\mathrm{R}})/14.75]\} \quad (1)$$

where t is the reaction time of the torrefaction in minutes, T_{H} the reaction temperature in °C, and T_{R} the reference temperature, most often of 100 °C.

2.3. Analysis of chemical components in the biomass

The chemical components in the biomass were analyzed using the TAPPI test method (1992). Insoluble and soluble lignins (Klason lignin) were determined by sulfuric acid treatment. Acid-soluble lignin content was determined by UV-spectrometry at 205 nm. Moisture content (T 207 om-88), holocellulose (T 249, Wise method) and Klason lignin (T 222-om-88) of torrefied biomass was compared with the raw material.

2.4. Elemental analysis and energy value of biomass

The moisture content of torrefied biomass was performed using the oven-dry method. The gross energy value based on dry weight at constant volume was determined. The samples were weighed, and ranged from 0.5 to 0.6 g; combustion was performed in a Parr 6400 automated isoperibol bomb calorimeter. The ash content was determined by burning the oven-dried sample (1 g) in a platinum crucible in a muffle furnace model at 575 ± 25 °C. The nitrogen, carbon and hydrogen contents of the torrefied biomass were assessed via direct combustion and thermal conductivity detection by using an elemental analyzer (Thermo EA 1112A, USA). In brief, the sample was dropped into a hot furnace and flushed with pure oxygen for very rapid combustion, forming combustion by-products (CO_2, H_2O, NO_x and N_2), and then passed through the furnace filter and thermoelectric cooler for subsequent collection in a ballast apparatus. The gases collected in the ballast were mixed, and a small aliquot was then used for further conversion of the gases. The remaining aliquot that was reduced was evaluated using the

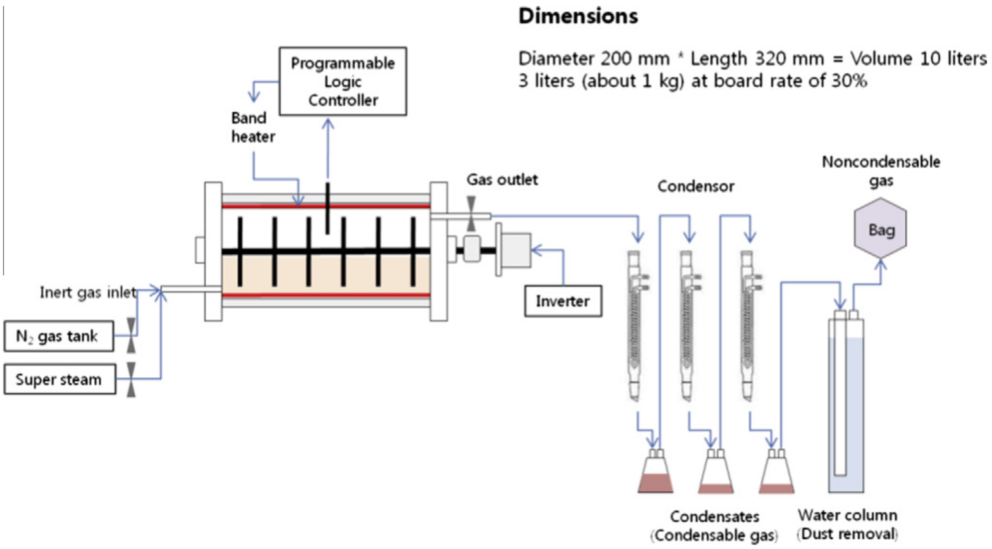
Dimensions
Diameter 200 mm * Length 320 mm = Volume 10 liters
3 liters (about 1 kg) at board rate of 30%
Programmable Logic Controller
Band heater
Gas outlet
Condensor
Noncondensable gas
Bag
Inverter
Inert gas inlet
N₂ gas tank
Super steam
Condensates (Condensable gas)
Water column (Dust removal)

Fig. 1. Design of torrefaction reactor used in this study.

thermal conductivity cell for nitrogen, carbon and hydrogen contents. The oxygen content was determined by elemental analyzer (Thermo Quest EA-1110, Italy). All experiments were performed in triplicate, according to the quality standard of the wood pellet (Korea Forest Service, 2009), and a mean value was reported.

2.5. Surface response analysis

A 2^2 factorial design was used for optimizing the torrefaction of mixed softwood to improve its energy density for wood pellet production. Time (X_1, 20–80 min) and reaction temperature (X_2, 220–280 °C) were chosen as the independent variables. Energy value, weight loss of biomass, and energy yield were used as the dependent output variables. Second-degree polynomials were calculated using Design-Expert Version 8.0.1 software (Stat-Ease Inc., USA) to estimate the response of the dependent variables.

Factors from each run were appropriately coded to real independent variables x_1 and x_2 for the statistical model as shown in Table 1. The independent variables were [(condition of the run–condition at the central point)/step change of the factor]. Therefore, x_1 corresponded to [(time – 50)/20], and x_2 was [(temperature – 250)/20].

3. Results and discussion

3.1. Chemical properties of torrefied biomass

The change in the chemical components of torrefied biomass is shown in Table 2. Holocellulose remaining in the torrefied biomass was thermally decomposed by torrefaction. Therefore, the holocellulose content decreased as the SF increased, but lignin was not degraded under the torrefaction conditions used in this study (Fig. 2). In particular, the thermal degradation of hemicelluloses and the release of the volatile components of biomass occurred at temperatures below 250 °C (Di Blasi and Lanzetta, 1997; Milosavljevic and Suuberg, 1995). At high SF, most of the hemicelluloses and some of the cellulose were thermally degraded during torrefaction. As a result, lignin accumulated in the torrefied biomass, and its amount increased with the SF. This result is very similar to that stated in the report by Phanphanich and Mani (2010). High lignin contents in torrefied biomass are associated with high energy density due to the ether and carbon–carbon linkages in lignin, which has a higher energy than the C—O or C—H bonds (Lehtikangas, 2001; Shafizadeh et al., 1976). Therefore, high lignin content in torrefied biomass directly results in a higher energy value. The moisture content in the biomass decreased from 6.76 to 2.86 depending on the SF. This is indicative of the reduction in hydroxyl groups during torrefaction. Ash contents were slightly increased as the SF increased. This might be due to the mass loss during torrefaction. The condensed liquids from the 11 torrefactions were analyzed by GC/MS (data not shown). Most of the components containing the condensed liquids were degraded products from hemicellulose, such as acetic acid, furfural, and formic acid.

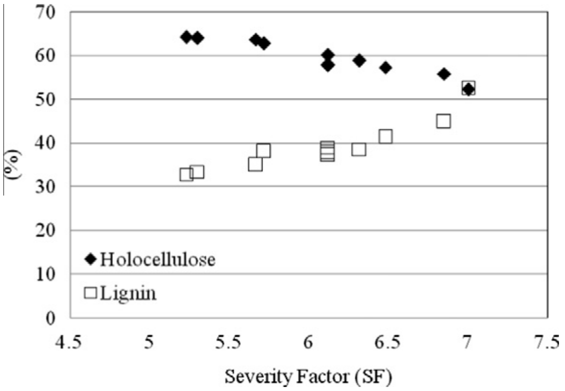

Fig. 2. Holocellulose and lignin contents in the biomass after torrefaction over a range of SF values.

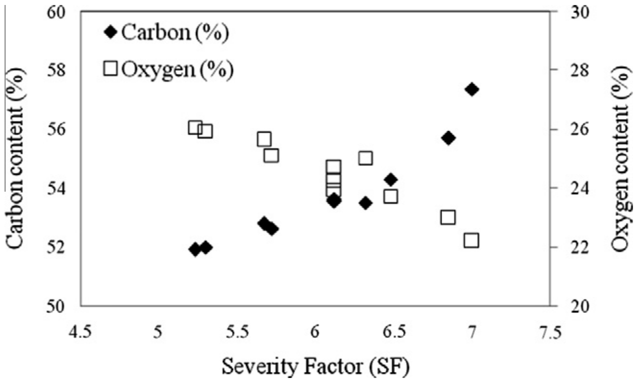

Fig. 3. Carbon and oxygen contents in the biomass after torrefaction over a range of SF values.

The elemental analysis of the raw biomass and the torrefied biomass is shown in Table 2. Torrefaction of the mixed softwood resulted in a higher carbon and low oxygen content as the SF increased (Fig. 3), resulting in decreased H/C and O/C ratios. This is due to the release of volatile compounds rich in hydrogen and oxygen, such as water and carbon dioxide. Torrefied biomass is believed to have a higher energy value (Bergmen et al., 2005, Prins et al., 2006). However, the nitrogen content remained almost constant.

Table 2
Chemical compositions and elemental analysis of the torrefied biomass.

SF	Holocellulose (%)	Total lignin[a] (%)	Moisture content (%)	Ash (%)	C (%)	H (%)	O (%)	N (%)
5.23	64.11 ± 1.44	32.79 ± 1.48	4.25 ± 0.56	0.99 ± 0.02	51.93 ± 0.13	6.53 ± 0.01	26.07 ± 0.06	0.41 ± 0.01
5.30	63.94 ± 1.39	33.38 ± 1.41	3.95 ± 1.08	0.56 ± 0.01	52.00 ± 0.06	6.65 ± 0.02	25.95 ± 0.05	0.40 ± 0.02
5.67	63.65 ± 1.94	34.91 ± 1.38	4.96 ± 1.00	0.87 ± 0.02	52.81 ± 0.19	6.28 ± 0.03	25.65 ± 0.05	0.44 ± 0.02
5.72	62.75 ± 1.43	38.04 ± 1.42	3.45 ± 0.82	0.96 ± 0.02	52.63 ± 0.09	6.54 ± 0.04	25.12 ± 0.06	0.40 ± 0.02
6.12	57.80 ± 1.29	37.78 ± 1.30	3.28 ± 1.25	0.80 ± 0.02	53.57 ± 0.14	6.14 ± 0.05	24.27 ± 0.06	0.43 ± 0.06
6.12	60.04 ± 1.29	37.16 ± 1.30	3.28 ± 1.02	0.76 ± 0.01	53.58 ± 0.07	6.24 ± 0.02	24.74 ± 0.07	0.40 ± 0.01
6.12	57.79 ± 1.23	38.92 ± 1.23	3.39 ± 1.08	0.78 ± 0.00	53.64 ± 0.06	6.19 ± 0.02	23.96 ± 0.07	0.43 ± 0.02
6.32	58.79 ± 1.37	38.50 ± 1.39	3.00 ± 0.67	0.84 ± 0.02	53.51 ± 0.10	6.48 ± 0.03	25.03 ± 0.05	0.41 ± 0.03
6.48	57.24 ± 1.23	41.31 ± 1.24	3.02 ± 1.11	0.83 ± 0.01	54.30 ± 0.09	6.32 ± 0.04	23.73 ± 0.06	0.39 ± 0.02
6.85	55.79 ± 1.30	44.78 ± 1.31	3.07 ± 1.03	1.32 ± 0.01	55.71 ± 0.37	6.07 ± 0.05	23.00 ± 0.09	0.42 ± 0.05
7.00	52.27 ± 1.39	52.45 ± 1.38	2.86 ± 0.68	1.26 ± 0.05	57.36 ± 0.18	6.06 ± 0.04	22.21 ± 0.07	0.39 ± 0.03
Raw material	75.00 ± 1.27	30.43 ± 1.57	6.76 ± 0.82	0.74 ± 0.01	50.79 ± 0.16	6.46 ± 0.03	27.20 ± 0.07	0.44 ± 0.03

[a] Total lignin = (acid-insoluble lignin) + (acid-soluble lignin).

3.2. Analysis of energy value and weight loss based on SF

Energy value and weight loss in the biomass over a range of SF values are shown in Table 3 and Fig. 4. The experimental energy value and weight loss increased with SF. This result is in agreement with the report of Medic et al. (2011). These factors are the major determinant in the energy density of biomass: their value slowly increased as the SF increased up until 6.12, then dramatically increased as the SF further increased from 6.12 to 7.0. This was more due to the removal of moisture and volatile compound remaining in the biomass, than to degradation of major compounds of biomass such as cellulose and lignin. It is similar to the change in carbon content in biomass during torrefaction.

The energy yield started decreasing above a SF of 6.12. The highest energy yield was obtained under mild torrefaction conditions. The energy yield showed an inverse proportion to the energy value and weight loss based on the SF.

3.3. Response surface analysis for weight loss, energy value and energy yield

The energy value, weight loss, and energy yield obtained from biomass depended on the torrefaction conditions listed in Table 3. The energy value ranged from 19.31 to 22.12 MJ/kg depending on the torrefaction conditions. This implies that the energy content in the torrefied biomass increased by 4–19% as compared to the untreated biomass. The increase in the energy value of biomass during torrefaction was comparable with that obtained in other similar studies for agricultural residues and wood chips (Phanphanich and Mani, 2010; Pimchuai et al., 2010; Prins et al., 2006). The energy value was affected more by the reaction time than by the temperature, as shown in Fig. 5. In particular, the energy value significantly increased with a reaction time of over 70 min, regardless of the torrefaction temperature. The highest energy value was 22.12 MJ/kg for the biomass torrefied at 280 °C for 50 min, corresponding to a weight loss of 33.53%. Based on the energy value, the response surface determined the optimal torrefaction condition to yield the highest energy value. The modeling results are provided in Fig. 5. A three-dimensional plot shows the predicted peak in energy value at the optimum condition. Y_1 is the predicted energy value (MJ/kg). The results of the analysis of variance (ANOVA) in Eq. (2) are presented in Table 4, where x_1 and x_2 denote reaction time and temperature. This model was significant at the 99% confidence level, and the *p*-values for temperature and reaction time were close to 0.

Table 3
Energy values and weight loss of biomass by torrefaction.

SF	Energy value (MJ/kg)	Weight loss[a] (%)	Mass yield[b] (%)	Energy yield (%)
5.23	19.38 ± 0.06	2.73	97.27	101.65
5.30	19.31 ± 0.04	4.83	95.17	99.11
5.67	19.88 ± 0.03	7.51	92.49	99.18
5.72	19.88 ± 0.19	8.11	91.89	98.51
6.12	19.82 ± 0.12	10.23	89.77	95.98
6.12	19.87 ± 0.09	8.98	92.02	97.54
6.12	19.88 ± 0.10	9.62	90.38	96.91
6.32	20.25 ± 0.15	12.87	87.13	95.16
6.48	20.47 ± 0.15	16.49	83.51	92.20
6.85	21.00 ± 0.07	23.23	76.77	86.95
7.00	22.12 ± 0.14	33.53	66.47	79.31
Raw material	18.54 ± 0.09	–	100	100

[a] Weight loss (%) = {(mass of untreated biomass − mass of torrefied biomass)/mass of untreated biomass}*100.
[b] Mass yield (%) = 100 − weight loss of biomass.

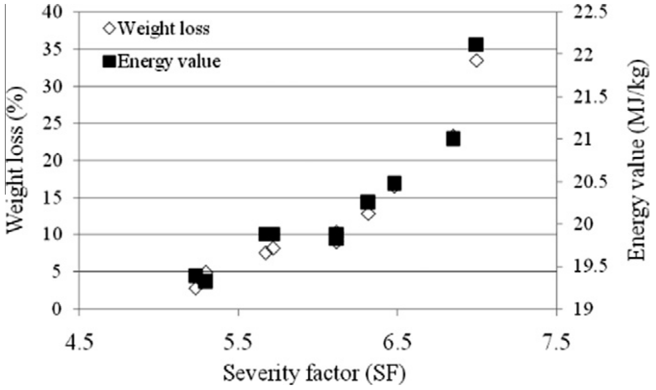

Fig. 4. Comparison of weight loss and energy value obtained from torrefied biomass depending on severity factors.

$$Y_1 = 4964.33 + 192.70x_1 + 50.94x_2 - 2.5x_1x_2 + 96.46x_1^2 + 10.46x_2^2 \quad (2)$$

The weight loss of the biomass during torrefaction is shown in Fig. 5. According to the model, temperature and time squared were the only significant parameters (data not shown). The strength of the effect of these two significant process parameters on the weight loss of biomass is revealed more clearly by the surface plot. Reaction temperature has an impact on biomass weight loss during torrefaction. However it has no effect on the energy value. This might be due to the removal of water and volatile compounds present in the biomass as an initial reaction of thermo-degradation of the biomass. The temperature ranged from 220 to 280 °C in this study. At this range, most of the hemicelluloses and some of the cellulose and lignin content in the biomass could be degraded during torrefaction. Therefore, more time was necessary to increase the weight loss of biomass at the given temperature range. Weight loss during torrefaction highly depended on reaction time, rather than on temperature. Table 4 shows the ANOVA of the quadratic model adjustment, where the total error was classified into lack of fit and pure error. The *F* value estimated using the experimental data corresponded to the total residual and lack-of-fit values, respectively, and was lower than the tabular *F* value. This indicates that the models were significant in the region studied. For both models, the *p*-value was 0.0025, which shows that the models were strongly significant at the 99% confidence level. The model calculated for the weight loss of biomass (Y_2) is provided in Eq. (3).

$$Y_2 = 9.61 + 9.87x_1 + 2.02x_2 + 1.01x_1x_2 + 3.94x_1^2 + 0.12x_2^2 \quad (3)$$

The results of the energy yield are shown in Fig. 6. They were calculated from the mass yield and energy value using Eq. (4), and expressed as a percentage of the energy content of untreated dry biomass: $m_{\text{torrefied}}$ denotes dry mass of torrefied biomass; m_{initial}, dry mass of untreated biomass; $E_{\text{torrefied}}$, specific energy content of biomass after torrefaction; and E_{initial}, specific energy content of biomass before torrefaction.

$$\text{Energy yield } (\%) = (m_{\text{torrefied}}/m_{\text{initial}}) \times (E_{\text{torrefied}}/E_{\text{initial}}) \quad (4)$$

The energy yield per raw biomass indicates the total energy preserved in the torrefied biomass. It was highest when the difference between the weight loss and energy value was high. Reaction time had the highest impact on the energy yield of torrefied biomass, while the effect of temperature was considerable lower. Energy yield was fitted to the response surface model provided in Eq. (5), in order to analyze the effect of the torrefaction factors on energy yield. As illustrated in Table 4, Eq. (5) shows in good correlation with the actual data as justified by the relatively high *R*-squared value.

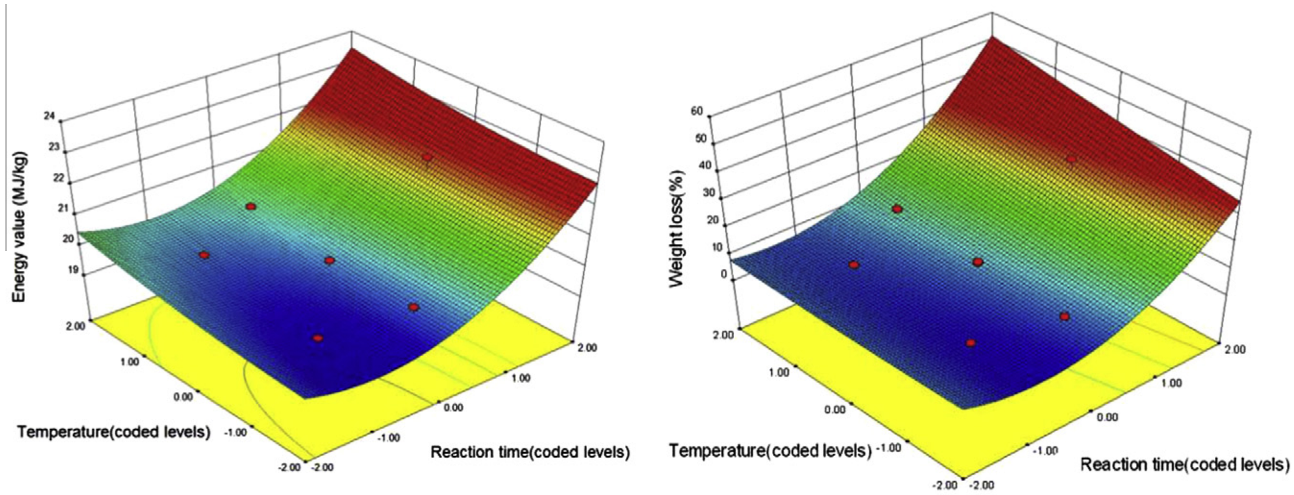

Fig. 5. Response surface and contour plot of temperature vs. reaction time on the energy value and weight loss during torrefaction.

Table 4
Analysis of variance (ANOVA) for the adjusted model for the energy value, weight loss and energy yield of biomass during torrefaction.

Source	Sum of squares	Degrees of freedom	Mean square	*F*-value	*p*-value (Prob. > *F*)
Energy value					
Model	372400	5	74472.34	12.33	0.0077
Residual	30195.19	5	6039.04		
Lack of fit	30086.52	3	10028.84	184.58	0.0054
Purr error	108.67	2	54.33		
Corrected total	402600	10			
Time	297100	1	297100	49.19	0.0009
Temp.	20759.26	1	20759.26	3.44	0.1229
Time × Temp.	25	1	25	0.004	0.9512
$(\text{Time})^2$	52541.42	1	52541.42	8.70	0.0319
$(\text{Temp.})^2$	617.66	1	617.66	0.10	0.7620
					Adjusted *R*-square: 0.8500
Weight loss					
Model	757.95	5	151.99	20.13	0.0025
Residual	37.75	5	7.55		
Lack of fit	36.97	3	12.32	31.54	0.0309
Purr error	0.78	2	0.39		
Corrected total	797.71	10			
Time	629.02	1	629.02	83.31	0.0003
Temp.	32.61	1	32.61	4.32	0.0923
Time × Temp.	4.12	1	4.12	0.55	0.4933
$(\text{Time})^2$	87.50	1	87.50	11.59	0.0192
$(\text{Temp.})^2$	0.076	1	0.076	0.010	0.9238
					Adjusted *R*-square: 0.9053
Energy yield					
Model	398.38	5	79.68	18.17	0.0032
Residual	21.92	5	4.38		
Lack of fit	20.78	3	6.93	12.15	0.0770
Purr error	1.14	2	0.57		
Corrected total	420.30	10			
Time	322.31	1	322.31	73.53	0.0004
Temp.	11.92	1	11.92	2.72	0.1600
Time × Temp.	7.02	1	7.02	1.60	0.2614
$(\text{Time})^2$	49.98	1	49.98	11.40	0.0198
$(\text{Temp.})^2$	0.23	1	0.23	0.052	0.8294
					Adjusted *R*-square: 0.8880

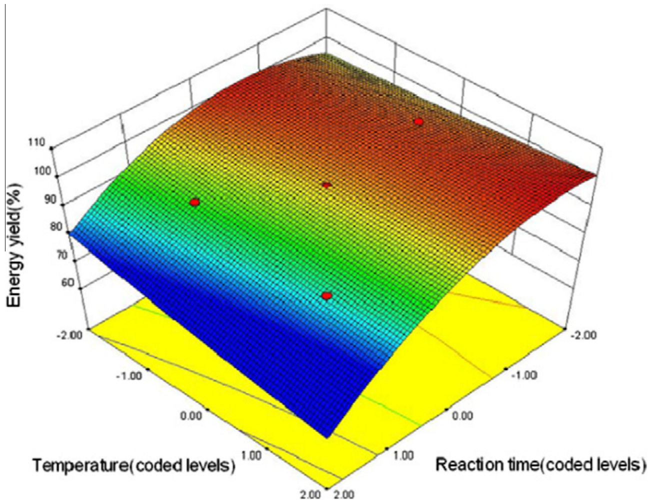

Fig. 6. Response surface and contour plot of temperature vs. reaction time on the energy yield during torrefaction.

The proposed model for measuring the energy yield was validated under the conditions determined by the statistical methodology. For that purpose, torrefaction was conducted at 230 °C for 30 min (the red[1] zone in Fig. 6). The values of energy value and weight loss of biomass estimated by Eqs. (2) and (3) were 19.30 MJ/kg and 4.81%, respectively. The result of the experimental assay was 19.31 MJ/kg and 4.83%, respectively, indicating that the models can mathematically represent energy value and weight loss of biomass by torrefaction.

The present study using the technique of response surface methodology (RSM) enables us to find the accurate values of the torrefaction condition for the maximum energy yield. Therefore, the optimal condition for the torrefaction of mixed softwood is the red zone in Fig. 6 which can be used to produce high energy density fuels when the weight loss of biomass by torrefaction is considered.

$$Y_3 = 96.80 - 6.35x_1 - 1.22x_2 - 1.33x_1x_2 - 2.98x_1^2 + 0.20x_2^2 \qquad (5)$$

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[1] For interpretation of color in Fig. 6, the reader is referred to the web version of this article.

4. Conclusions

The optimal condition for the torrefaction of mixed softwood was investigated with respect to the reaction temperature and time. While the reaction time had a strong impact on the energy yield of torrefied biomass, the effect of temperature was considerably lesser, under the torrefaction conditions used in this study. At a low SF, torrefaction had a significant effect on the degradation of hemicelluloses, but not on cellulose or lignin. Therefore, when the weight loss of biomass by torrefaction is considered, introducing low SF values for mixed softwood is appropriate for the torrefaction of biomass for the in production of high energy density fuels.

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